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8/20/09

Ravi Sanga/R10/USEPA/US
08/20/2009 03:26 PM

To Susan McGroddy <SusanM@windwardenv.com>

cc "Dave Schuchardt (Dave.Schuchardt@Seattle.Gov)" <Dave.Schuchardt@Seattle.Gov>, dan berlin <dberlin@anchorenv.com>, "Debra Williston (debra.williston@kingcounty.gov)" <debra.williston@kingcounty.gov>, "Debra Williston (b) (6) Gary Pascoe (b) (6) >, Doug Hotchkiss <hotchkiss.d@portseattle.org>, "Jeff Stern (jeff.stern@kingcounty.gov)" <jeff.stern@kingcounty.gov>, Lisa Gagnon <lgagnon@anchorenv.com>, Lon Kissinger/R10/USEPA/US@EPA, Nancy Judd <NancyJ@windwardenv.com>, "Pete Rude (pete.rude@seattle.gov)" <pete.rude@seattle.gov>, Scott Becker <sbecker@integral-corp.com>, Susan McGroddy <SusanM@windwardenv.com>, "twang@anchorenv.com" <twang@anchorenv.com>, Lon Kissinger/R10/USEPA/US@EPA

bcc

Subject Re: Additional analyses for Fish and Crab tissues

Susie -- EPA has reviewed the proposal below and the only modification that should be made is that three supercomposites must be run for the mussels so that 95% UCLs can be generated.

Please also provide more information on how the mussel samples will be created.

Other than that the analysis can proceed as planned.

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Susan McGroddy Ravi, 08/18/2009 03:52:44 PM

Susan McGroddy <SusanM@windwardenv.com>
08/18/2009 03:51 PM

To Ravi Sanga/R10/USEPA/US@EPA, Lon Kissinger/R10/USEPA/US@EPA

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Subject Additional analyses for Fish and Crab tissues

Ravi,

Here is our proposal for the additional analyses of the fish (English sole, shiners), crab, mussels and geoducks for PAHs, pentachlorophenol (PCP) and bis-2ethylhexyl phthalate (BEHP). The holding times for all samples except the geoducks expire next week. We need a response from EPA by COB on Thursday with regard to this proposal so the lab can be instructed to initiate the analyses within the holding times.

Thanks.
Susie

PAHs

All existing composite samples with sufficient mass will be analyzed for PAHs.

PAHs will be analyzed by EPA-8270SIM at CAS with reporting limits consistent with the previous reanalyses for clam and rockfish tissues and the results of the LDW analyses.

Pentachlorophenol and BEHP

For geoducks, the samples with sufficient mass remaining will be analyzed for PCB and BEHP (edible meat samples: GD-02,GD-03, GD-04, GD-07, GD-10, gut ball samples: GB-comp1 and GB-comp2)

For fish and crabs, the supercomposite samples that were created for the analysis of PCB congeners and dioxins and furans will be analyzed for PCP and BEHP. These chemicals were rarely detected in the LDW fish and crab tissue samples (Table 1). The original analysis of these tissues was not a high resolution analysis and the reporting limits were elevated. Additional high resolution analyses were conducted and these chemicals were confirmed as non-detected at lower RLs (1000 times lower than original RLs). The memo detailing the high resolution analyses is attached to this e-mail. The preliminary results for the EW clam samples were reviewed and PCP was detected in two of the ten samples analyzed and BEHP was not detected in any of the EW clam samples.

The EPC for these chemicals can be calculated from the supercomposites in the same way the TEQ EPC will be.

Table 1. Detection frequency for BEHP and PCP in LDW fish and crab tissues

Tissue	BEHP detection frequency	PCP detection frequency
English sole – whole body	0/21	1/21
English sole – fillet	2/7	0/7
Starry flounder – whole body	0/3	0/3
Starry flounder - fillet	0/1	0/1
Pacific staghorn sculpin	0/24	3/24
shiner surfperch	5/24	3/24[a]
pile perch	0/1	0/1
Dungeness crab – edible meat	0/7	0/1
Dungeness crab – hepatopancreas	0/3	0/3

Slender crab-edible meat	0/12	0/12
Slender crab – hepatopancreas	0/4	0/4

[a] Two samples with detected results were submitted for high resolution analysis and the detections were not confirmed.

For mussels, we will create a super composite sample that combines equal mass from all the existing mussel composites. This composite will represent 1,100 mussels collected throughout the waterway.

BEHP and PCP will be analyzed by EPA-8270SIM at ARI with reporting limits consistent with the previous reanalyses for clam and rockfish tissues and the results of the LDW analyses

Fish-crab_data_addendum.doc

Lower Duwamish Waterway Group

Port of Seattle / City of Seattle / King County / The Boeing Company

Lower Duwamish Waterway Remedial Investigation

FISH AND CRAB TISSUE DATA REPORT ADDENDUM: BIS(2-ETHYLHEXYL)PHTHALATE AND PENTACHLOROPHENOL RE-ANALYSES FINAL

For submittal to

The US Environmental Protection Agency
Region 10
Seattle, WA

The Washington State Department of Ecology
Northwest Regional Office
Bellevue, WA

March 29, 2006

Prepared by: Wind/Ward environmental LLC

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Table of Contents

Acronyms

Acronym	Definition
ARI	Analytical Resources, Inc.
Axys	Axys Analytical Services, Ltd.
CAS	Columbia Analytical Services
EPA	US Environmental Protection Agency
GC/ECD	gas chromatography/electron capture detection
GC/MS	gas chromatography/mass spectrometry
LCS	laboratory control sample
LDC	Laboratory Data Consultants, Inc.
LDW	Lower Duwamish Waterway
MS/MSD	matrix spike/matrix spike duplicate
PCP	pentachlorophenol
QA	quality assurance
QAPP	quality assurance project plan
QC	quality control
RPD	relative percent difference
RI	remedial investigation
RL	reporting limit
SDG	sample delivery group
SVOC	semivolatile organic compound
Windward	Windward Environmental LLC
ww	wet weight

1.0 Introduction

This data report is an addendum to the *Lower Duwamish Waterway Remedial Investigation Data Report: Fish and crab tissue collection and chemical analyses* (Windward 2005). It provides the results of chemical analyses of archived tissue samples that were previously collected as part of the Lower Duwamish Waterway (LDW) Phase 2 remedial investigation (RI). A subset of the archived fish and crab tissue samples were submitted for additional analyses of bis(2-ethylhexyl)phthalate and pentachlorophenol (PCP) to achieve lower reporting limits (RLs) than those achieved in the original analyses. Additional cleanup steps and an alternate analytical method (for PCP) were employed to minimize matrix interferences, resulting in more sensitive instrument response and lower RLs. The re-analysis results for these two analytes will be selected for use in the Phase 2 RI because of the greater sensitivity of the re-analysis methods for these analytes.

This report is organized into sections addressing sample selection and laboratory analyses, chemical analysis results, data validation results, and references. The text is supported by the following appendices:

- Appendix A – Data management
- Appendix B – Data validation report
- Appendix C – Laboratory form 1s
- Appendix D – Chain of custody forms

2.0 Sample Selection and Laboratory Analyses

Tissue samples were collected from various areas within the LDW (Figure 2-1) in 2004 as part of the Phase 2 RI sampling and initially analyzed by Columbia Analytical Services (CAS) for semivolatile organic compounds (SVOCs) using US Environmental Protection Agency (EPA) Method 8270-SIM. The results of those analyses and information regarding the tissue sampling and processing, chemical analysis, and data validation were presented in the *Lower Duwamish Waterway Remedial Investigation Data Report: Fish and crab tissue collection and chemical analyses* (Windward 2005). In several samples, bis(2-ethylhexyl)phthalate and PCP were not detected, but RLs were elevated.

Subsamples of the original homogenized tissue samples analyzed by CAS were archived frozen by CAS in the event that additional analyses might be necessary. Forty-nine of those subsamples (Table 2-1) were selected for re-analysis of bis(2-ethylhexyl)phthalate and PCP if the sample had an elevated RL in the original

result. Archived tissue homogenates were shipped from CAS to Analytical Resources, Inc. (ARI), for analysis by alternate analytical methods to achieve lower RLs.

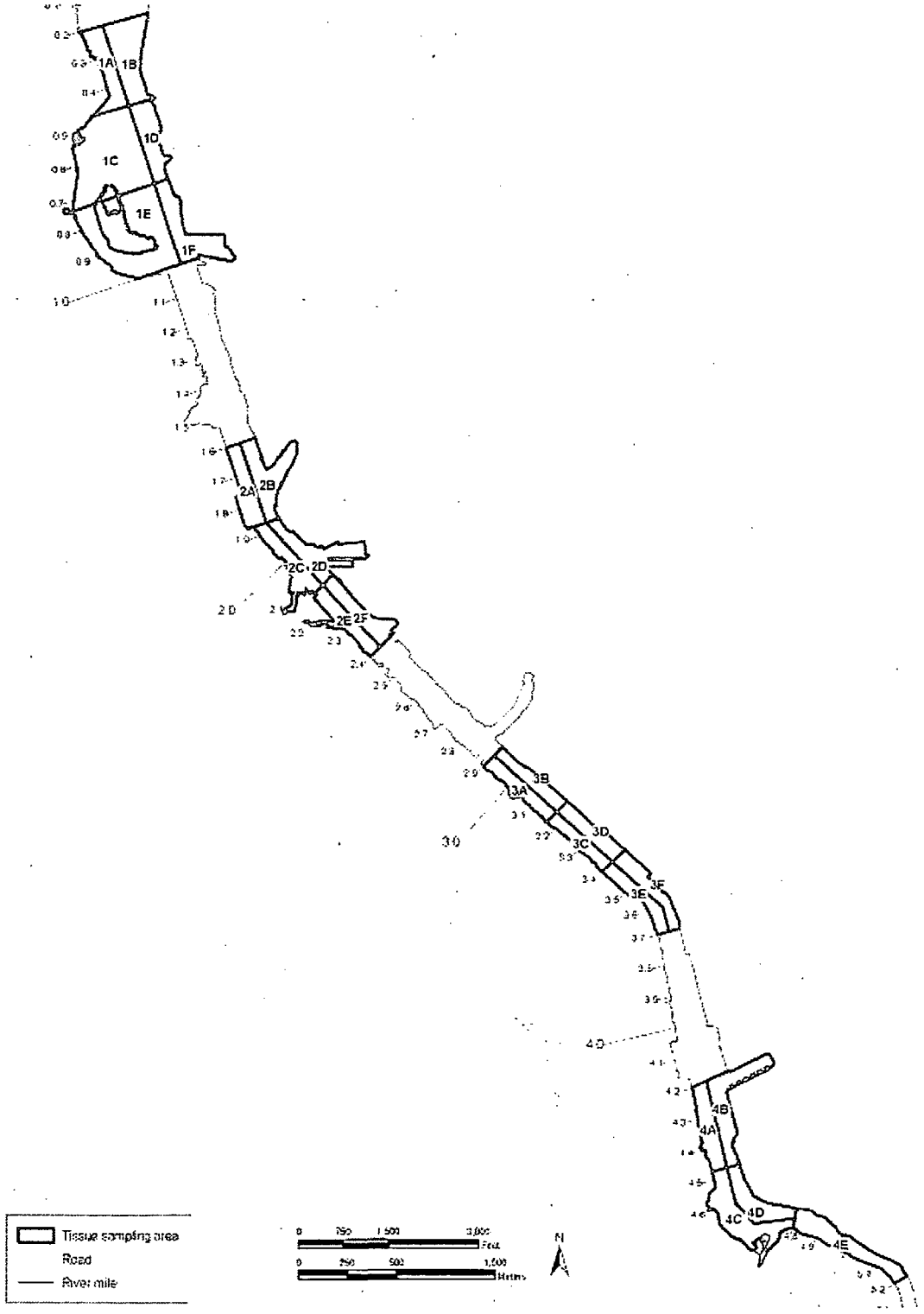

Figure 2-1. LDW fish and crab tissue collection areas

Table 2-1. Numbers of samples selected for bis(2-ethylhexyl)phthalate and PCP re-analysis

Species	Fillet Samples	Number of Samples	
		Whole Body	Other Samples
English sole	5	11	0
Pile perch	1	0	0
Striped perch	1	0	0
Shiner surfperch	0	4	0
Starry flounder	1	3	0
Crab – hepatopancreas	0	0	7
Crab – edible meat	0	0	16
Total	8	18	23

3.0 Results of Chemical Analyses

The analytical methods used by ARI are presented in Section 3.1. The results of the bis(2-ethylhexyl)phthalate and PCP re-analyses conducted on the 49 fish and crab tissue samples are presented in Section 3.2.

3.1 Analytical Methods

This section presents the analytical methods used by ARI for the fish and crab tissue samples. The analytical methods adhered to the most recent EPA quality assurance (QA) and quality control (QC) guidelines and analysis protocols. The methods of chemical analysis are identified in Table 3-1. All samples underwent a silica gel cleanup to minimize matrix interferences.

Table 3-1. Analytical methods for fish and crab tissue analyses by ARI

Parameter	Unit	Method	Reference
Bis(2-ethylhexyl)phthalate	µg/kg ww	GC/MS	EPA 8270D
Pentachlorophenol	µg/kg ww	GC/ECD	EPA 8041

ww – wet weight

GC/ECD – gas chromatography/electron capture detection

GC/MS – gas chromatography/mass spectrometry

3.2 Results

This section presents the results of the re-analyses for bis(2-ethylhexyl)phthalate (Section 3.2.1) and PCP (Section 3.2.2) in fish and crab tissue samples. Table 3-2 lists both the results of the re-analyses by ARI and the previous results reported for these compounds by CAS in the *Lower Duwamish Waterway Remedial Investigation Data Report: Fish and crab tissue collection and chemical analyses* (Windward 2005). All results were reported by the laboratories to two significant figures.

Table 3-2. Original (CAS) and re-analysis (ARI) results for bis(2-ethylhexyl)phthalate and PCP

Sample ID	Unit	Concentration			
		Bis(2-ethylhexyl)phthalate		Pentachlorophenol	
		CAS	ARI	CAS	ARI
Dungeness crab – edible meat					
LDW-T1-M-DC-EM-comp-1	µg/kg ww	7,200 UJ	130 UJ	5,800 U	7.4 U
LDW-T1-M-DC-EM-comp-2	µg/kg ww	7,200 UJ	67 U	5,700 U	3.3 U
LDW-T1-M-DC-EM-comp-3	µg/kg ww	7,200 UJ	66 UJ	5,700 U	3.3 U
LDW-T3-M-DC-EM-comp-1	µg/kg ww	7,200 UJ	67 U	5,700 U	3.3 U
LDW-T3-M-DC-EM-comp-2	µg/kg ww	7,200 UJ	67 U	5,700 U	3.3 U
LDW-T3-M-DC-EM-comp-3	µg/kg ww	7,200 UJ	66 U	5,700 U	3.3 U
LDW-T4-M-DC-EM-comp-1	µg/kg ww	7,200 U	67 U	5,700 U	6.7 U
Dungeness crab – hepatopancreas					
LDW-T1-M-DC-HP-comp-1	µg/kg ww	7,200 UJ	66 U	5,800 U	3.3 U
LDW-T3-M-DC-HP-comp-1	µg/kg ww	7,200 UJ	66 U	5,700 U	3.3 U
LDW-T4-M-DC-HP-comp-1	µg/kg ww	7,200 U	130 UJ	5,700 U	4.6 U
English sole – fillet					
LDW-T1-M-ES-FL-comp-2	µg/kg ww	7,200 U	67 UJ	5,800 U	3.3 U
LDW-T2-M-ES-FL-comp-1	µg/kg ww	7,200 U	67 UJ	5,700 U	3.3 U
LDW-T2-M-ES-FL-comp-2	µg/kg ww	7,200 U	67 UJ	5,800 U	3.3 U
LDW-T3-M-ES-FL-comp-1	µg/kg ww	7,200 U	67 U	5,700 U	6.7 U
LDW-T4-M-ES-FL-comp-1	µg/kg ww	7,200 U	130 UJ	5,800 U	8.6 U
English sole – whole body					
LDW-T2-M-ES-WB-comp-4	µg/kg ww	7,200 U	67 UJ	5,700 U	2.3 J
LDW-T2-M-ES-WB-comp-5	µg/kg ww	7,200 U	66 UJ	5,800 U	3.3 U
LDW-T2-M-ES-WB-comp-6	µg/kg ww	7,200 U	67 UJ	5,700 U	1.9 J
LDW-T3-M-ES-WB-comp-1	µg/kg ww	7,200 U	66 UJ	5,800 U	3.3 U
LDW-T3-M-ES-WB-comp-2	µg/kg ww	7,200 U	66 UJ	5,800 U	3.3 U
LDW-T3-M-ES-WB-comp-3	µg/kg ww	7,200 U	67 UJ	5,700 U	6.7 U
LDW-T3-M-ES-WB-comp-4	µg/kg ww	7,200 U	83 U	5,700 U	4.1 U
LDW-T3-M-ES-WB-comp-5	µg/kg ww	7,200 U	66 U	5,700 U	3.3 U
LDW-T3-M-ES-WB-comp-6	µg/kg ww	7,200 UJ	67 UJ	5,800 U	3.3 U
LDW-T4-M-ES-WB-comp-1	µg/kg ww	7,200 UJ	66 U	5,800 U	1.5 J
LDW-T4-M-ES-WB-comp-3	µg/kg ww	7,200 UJ	67 U	5,700 U	1.1 J
Pile perch – fillet					
LDW-M-M-PP-FL-comp-1	µg/kg ww	7,200 U	67 U	5,800 U	6.7 U
Slender crab – edible meat					
LDW-T2-M-SC-EM-comp-1	µg/kg ww	7,200 U	67 U	5,800 U	3.3 U
LDW-T2-M-SC-EM-comp-2	µg/kg ww	7,200 U	130 UJ	5,700 U	3.7 U

Sample ID	Unit	Concentration			
		Bis(2-ethylhexyl)phthalate		Pentachlorophenol	
		CAS	ARI	CAS	ARI
LDW-T2-M-SC-EM-comp-3	µg/kg ww	7,200 U	90 U	5,700 U	3.3 U
LDW-T2-M-SC-EM-comp-4	µg/kg ww	7,200 U	66 U	5,800 U	3.3 U
LDW-T2-M-SC-EM-comp-5	µg/kg ww	7,200 U	66 U	5,800 U	3.3 U
LDW-T2-M-SC-EM-comp-6	µg/kg ww	7,200 U	66 U	5,700 U	3.3 U
LDW-T3-M-SC-EM-comp-1	µg/kg ww	7,200 U	72 U	5,800 U	7.2 U
LDW-T3-M-SC-EM-comp-2	µg/kg ww	7,200 U	67 UJ	5,800 U	3.3 U
LDW-T3-M-SC-EM-comp-3	µg/kg ww	7,200 U	66 U	5,700 U	3.3 U
Slender crab – hepatopancreas					
LDW-T1-M-SC-HP-comp-1	µg/kg ww	7,200 U	67 UJ	5,700 U	11 U
LDW-T2-M-SC-HP-comp-1	µg/kg ww	7,200 U	66 UJ	5,800 U	3.3 UJ
LDW-T2-M-SC-HP-comp-2	µg/kg ww	7,200 U	100 J	5,800 U	3.3 UJ
LDW-T3-M-SC-HP-comp-1	µg/kg ww	7,200 U	230 UJ	5,700 U	4.4 U
Starry flounder – fillet					
LDW-T4-M-SF-FL-comp-1	µg/kg ww	7,200 UJ	67 UJ	5,700 U	3.3 UJ
Starry flounder – whole body					
LDW-T4-M-SF-WB-comp-1	µg/kg ww	7,200 UJ	66 U	5,800 U	1.3 J
LDW-T4-M-SF-WB-comp-2	µg/kg ww	7,200 UJ	67 UJ	5,700 U	6.7 U
LDW-T4-M-SF-WB-comp-3	µg/kg ww	7,200 UJ	67 UJ	5,700 U	3.3 U
Striped perch – fillet					
LDW-M-M-SP-FL-comp-1	µg/kg ww	7,200 U	67 U	5,800 U	6.7 U
Shiner surfperch – whole body					
LDW-T3-A-SS-WB-comp-1	µg/kg ww	7,200 U	91 UJ	2,200 J	4.6 U
LDW-T3-B-SS-WB-comp-1	µg/kg ww	7,200 U	67 U	5,700 U	2.8 J
LDW-T3-C-SS-WB-comp-1	µg/kg ww	7,200 U	76 U	5,700 U	7.6 U
LDW-T3-D-SS-WB-comp-1	µg/kg ww	7,200 U	89 UJ	2,200 J	4.5 U

J – estimated concentration

U – not detected at reporting limit shown

UJ – not detected at estimated reporting limit shown

3.2.1 Bis(2-ethylhexyl)phthalate

The initial analyses conducted by CAS resulted in 49 non-detected results for bis(2-ethylhexyl)phthalate, with RLs of 7,200 µg/kg ww. The ARI re-analysis resulted in a single detection, at an estimated concentration of 100 µg/kg ww, in a slender crab hepatopancreas sample. Bis(2-ethylhexyl)phthalate was not detected by ARI in the remaining 48 samples, with RLs ranging from 66 to 230 µg/kg ww.

3.2.2 Pentachlorophenol

The initial analyses conducted by CAS resulted in 47 non-detected results for PCP, with RLs ranging from 5,700 to 5,800 µg/kg ww. PCP was detected by CAS in two shiner surfperch whole-body samples, both at estimated concentrations of 2,200 µg/kg ww. Neither of these detected results was confirmed by ARI's GC/ECD analyses; both samples were non-detect, one with an RL of 4.5 µg/kg ww and the other with an RL of 4.6 µg/kg ww. PCP was detected at low concentrations in 6 of the 47 samples originally reported as non-detect by CAS. Detected results included one shiner surfperch whole-body sample at an estimated concentration of 2.8 µg/kg ww, one starry flounder whole-body sample at an estimated concentration of 1.3 µg/kg ww, and four English sole whole-body samples at estimated concentrations ranging from 1.1 to 2.3 µg/kg ww. ARI also reported 43 non-detected results, with RLs ranging from 3.3 to 11 µg/kg ww.

4.0 Data Validation Results

Independent data validation of all results was conducted by Laboratory Data Consultants, Inc. (LDC). The following sections summarize the results of the validation but do not list every sample affected by qualification in this summary. Detailed information regarding every qualified sample is available in the complete data validation report in Appendix B.

4.1 Overall data quality

The tissue composite samples were analyzed by ARI in four sample delivery groups (SDGs). LDC conducted a full validation on one of the SDGs (IH52). All sample results that were not selected for full validation underwent a summary validation. The summary validation included a review of calibration and internal standard summary forms. Table 4-1 provides a summary of the number of samples in each SDG and the level of data validation. The percent of samples submitted for full validation for each analysis is consistent with the requirements of the quality assurance project plan (QAPP) (Windward 2004).

Table 4-1. Numbers of samples in each SDG and level of validation

SDG	Lab	Level of Validation	Number of Samples	
			Bis(2-ethylhexyl)phthalate	Pentachlorophenol
IH50	ARI	summary	10	10
IH51	ARI	summary	14	14
IH52	ARI	full	15	22
IM87	ARI	summary	10	3

The majority of the data were either not qualified or had "J" (estimate) qualifiers added as a result of the data validation. Based on the information reviewed, the

overall data quality was considered acceptable, as qualified for use in the Phase 2 RI. The results of the data validation are summarized (by analyte) in Sections 4.3 and 4.4.

4.2 Sample transport and holding times

Subsamples of the original tissue homogenates were archived frozen at CAS for a period of 222 to 230 days prior to their shipment to ARI. Samples were shipped frozen, and the chain-of-custody documents were reviewed for documentation of cooler temperatures. Temperatures inside the coolers were -15°C, meeting validation criteria. The subsamples can reasonably be expected to have remained frozen during the time in transit (24 hours) to ARI. Upon receipt at ARI, the samples were stored frozen until extraction, for 10 to 33 days. All re-analyses of the tissue samples were conducted within the 1-year maximum holding time for frozen samples, with the following exceptions.

ARI noted that there was insufficient sample volume for both bis(2-ethylhexyl)phthalate and PCP analyses for three samples, and a laboratory spiking error resulted in the consumption of the available sample volume for seven samples for bis(2-ethylhexyl)phthalate analysis. To compensate for these QA issues, additional archived subsamples were requested from Axys Analytical Services, Ltd. (Axys), where additional subsamples of the original homogenized samples had also been archived frozen. The chain-of-custody documents used by Axys for shipping to ARI were reviewed for documentation of the cooler temperature. The temperature inside the cooler was -36°C, meeting validation criteria. Samples were in transit for approximately 24 hours and can reasonably be expected to have remained frozen during shipping. The subsamples shipped from Axys were stored frozen at ARI for five days until extraction. As a result of this subsequent sample shipment, 10 samples were extracted 9 to 13 days past the 1-year maximum holding time specified in the QAPP (Windward 2004) for bis(2-ethylhexyl)phthalate, and three samples were extracted 9 to 13 days past the QAPP-specified 1-year maximum holding time for PCP. All analyses of the extracts were conducted within the maximum allowable 40-day extract holding time. The chemicals of concern were not detected in any of the samples that exceeded the 1-year maximum holding time for frozen samples, and all the results for these samples were UJ-qualified.

4.3 Bis(2-ethylhexyl)phthalate

Method blank results, internal standard recoveries, and matrix spike recoveries were the only sources of validation qualifiers for bis(2-ethylhexyl)phthalate; all other QC requirements were met.

Two method blanks contained bis(2-ethylhexyl)phthalate. As a result of the blank contamination, the one sample with detected bis(2-ethylhexyl)phthalate (LDW-T3-M-SC-HP-comp-1) was qualified as undetected (U), with an estimated RL of 230 µg/kg ww.

Low internal standard recoveries resulted in the UJ-qualification of bis(2-ethylhexyl)phthalate in 14 samples in SDGs IH50 and IH51.

In SDG IM87, the 79% relative percent difference (RPD) between the matrix spike and matrix spike duplicate was outside of QC limits (≤50%), resulting in the UJ-qualification of bis(2-ethylhexyl)phthalate in LDW-T4-M-SF-FL-comp-1. The matrix spike duplicate recovery (13%) and RPD (59%) in SDG IH52 were outside QC limits, resulting in the UJ-qualification of this chemical in sample LDW-T1-M-DC-EM-comp-3.

4.4 Pentachlorophenol

Surrogate and matrix spike recoveries were the only source of validation qualifiers for pentachlorophenol; all other QC requirements were met. High surrogate recoveries, ranging from 168 to 205%, resulted in the J-qualification of three samples for PCP.

In SDG IM87, the 59% RPD between the matrix spike and matrix spike duplicate was outside of QC limits (≤50%), resulting in the UJ-qualification of PCP in sample LDW-T4-M-SF-FL-comp-1.

5.0 References

Windward. 2004. Lower Duwamish Waterway remedial investigation. Quality assurance project plan: Fish and crab tissue collection and chemical analyses. Prepared for Lower Duwamish Waterway Group. Windward Environmental LLC, Seattle, WA.

Windward. 2005. Lower Duwamish Waterway remedial investigation. Data report: Fish and crab tissue collection and chemical analyses. Prepared for Lower Duwamish Waterway Group. Windward Environmental LLC, Seattle, WA.